BS EN 16711-2:2015

BSI Standards Publication

Textiles — Determination of metal content

Part 2: Determination of metals extracted by acidic artificial perspiration solution

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National foreword

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The UK participation in its preparation was entrusted to Technical Committee TCI/80, Chemical testing of textiles.

A list of organizations represented on this committee can be obtained on request to its secretary.

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Published by BSI Standards Limited 2015

ISBN 978 0 580 78748 5

ICS 59.060.01

Compliance with a British Standard cannot confer immunity from legal obligations.

This British Standard was published under the authority of the Standards Policy and Strategy Committee on 30 November 2015.

Amendments/corrigenda issued since publication

Date	Text affected

EUROPEAN STANDARD

NORME EUROPÉENNE

EUROPÄISCHE NORM

EN 16711-2

November 2015

ICS 59.060.01

English Version

Textiles - Determination of metal content - Part 2: Determination of metals extracted by acidic artificial perspiration solution

Textiles - Détermination de la teneur en métaux - Partie 2: Dosage des métaux extraits au moyen d'une solution de sueur artificielle acide

Textilien - Bestimmung von Metallen - Teil 2: Bestimmung von extrahierbaren Metallen mit saurer synthetischer Schweißlösung

This European Standard was approved by CEN on 5 September 2015.

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Contents

Page

European foreword

This document (EN 16711-2:2015) has been prepared by Technical Committee CEN/TC 248 "Textiles and textile products", the secretariat of which is held by BSI.

This European Standard shall be given the status of a national standard, either by publication of an identical text or by endorsement, at the latest by May 2016, and conflicting national standards shall be withdrawn at the latest by May 2016.

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EN 16711, *Textiles — Determination of metal content* is composed of the following parts:

— *Part 1: Determination of metals using microwave digestion*;

— *Part 2: Determination of metals extracted by acidic artificial perspiration solution.*

According to the CEN/CENELEC Internal Regulations, the national standards organizations of the following countries are bound to implement this European Standard: Austria, Belgium, Bulgaria, Croatia, Cyprus, Czech Republic, Denmark, Estonia, Finland, Former Yugoslav Republic of Macedonia, France, Germany, Greece, Hungary, Iceland, Ireland, Italy, Latvia, Lithuania, Luxembourg, Malta, Netherlands, Norway, Poland, Portugal, Romania, Slovakia, Slovenia, Spain, Sweden, Switzerland, Turkey and the United Kingdom.

1 Scope

This European Standard specifies a procedure for determination of antimony (Sb), arsenic (As), cadmium (Cd), chromium (Cr), cobalt (Co), copper (Cu), lead (Pb), mercury (Hg), nickel (Ni) in natural and man-made textiles, including coated fabrics and garment components (e.g. buttons, zips, etc.) after extraction with acidic artificial perspiration solution.

2 Normative references

The following documents, in whole or in part, are normatively referenced in this document and are indispensable for its application. For dated references, only the edition cited applies. For undated references, the latest edition of the referenced document (including any amendments) applies.

EN 1233, *Water quality — Determination of chromium — Atomic absorption spectrometric methods*

EN ISO 105-E04, *Textiles — Tests for colour fastness — Part E04: Colour fastness to perspiration (ISO 105-E04)*

EN ISO 3071, *Textiles — Determination of pH of aqueous extract (ISO 3071)*

EN ISO 3696, *Water for analytical laboratory use — Specification and test methods (ISO 3696)*

EN ISO 11885, *Water quality — Determination of selected elements by inductively coupled plasma optical emission spectrometry (ICP-OES) (ISO 11885)*

EN ISO 12846, *Water quality — Determination of mercury — Method using atomic absorption spectrometry (AAS) with and without enrichment (ISO 12846)*

EN ISO 15586, *Water quality — Determination of trace elements using atomic absorption spectrometry with graphite furnace (ISO 15586)*

EN ISO 17294-2, *Water quality — Application of inductively coupled plasma mass spectrometry (ICP-MS) — Part 2: Determination of 62 elements (ISO 17294-2)*

EN ISO 17852, *Water quality — Determination of mercury — Method using atomic fluorescence spectrometry (ISO 17852)*

ISO 8288, *Water quality — Determination of cobalt, nickel, copper, zinc, cadmium and lead — Flame atomic absorption spectrometric methods*

3 Principle

For determination of soluble metals, the textile specimen is cut into small pieces and is extracted with acidic artificial perspiration solution.

Analysis is performed with appropriate analytical techniques of atomic absorption and mass spectrometry (e.g. ICP-MS, ICP-OES, AAS, cold vapour atomic absorption spectrometry, etc.).

4 Apparatus

General laboratory equipment for analytical chemistry. In addition, the following is required:

4.1 Analytical balance, readability 0,000 1 g.

4.2 Erlenmeyer flask (100 ml) with stopper or other appropriate closable vessel.

4.3 pH meter with glass electrode according to EN ISO 3071.

4.4 Shaker, optional horizontal or circular shaker.

4.5 Heating source (37 ± 2) °C, e.g. water bath.

4.6 Filter, pore size of 0,45 µm.

5 Reagents

Unless otherwise specified, analytical grade chemicals shall be used.

5.1 Perspiration solution complying with EN ISO 105-E04.

Dissolve with water (5.4) in a 1 000 ml flask:

— 0,5 g L-histidine-monohydrochloride-1-hydrate;

— 5,0 g sodium chloride;

— 2,2 g sodium dihydrogenphosphate-2-hydrate.

Adjust the solution with diluted sodium hydroxide (5.3) or diluted hydrochloric acid (5.2) to pH (5,5 ± 0,2). Verify the compliance of the pH value using a pH electrode. Subsequently fill up with water (5.4) to the mark and homogenize the solution.

5.2 Hydrochloric acid, c(HCl) = 1 mol/l.

5.3 Sodium hydroxide, c(NaOH) = 1 mol/l.

5.4 Grade 2 water, complying with EN ISO 3696.

5.5 Metal standard solutions for calibration of the analytical systems complying with EN 1233, EN ISO 11885, EN ISO 12846, EN ISO 15586, EN ISO 17294-2, EN ISO 17852 or ISO 8288.

5.6 Nitric acid, ω (HNO_3) = 65 %.

5.7 Dilution solution, 1,2 ml nitric acid (5.6) is diluted with water (5.4) to 100 ml.

6 Test specimen sampling

Cut from the specimen, with a sharp pair of scissors made of noncorrosive material (preferably of ceramics) a representative sub-sample of approximately 1 g and record the mass to the nearest 0,1 mg. If the textile article is composed of several parts of products, such as knitted cuffs or lining, all kinds of materials have to be tested as a representative composite sample with equal parts of the materials. Select the number of parts that can be collected as a composite sample, in correlation with the detection and quantification limits of the instrumental equipment. Weigh out materials, which cannot be separated, as a composite sample. Consider also different colours of the material for sampling. Avoid any contact with metal articles, especially in wet conditions or with corrosive surfaces.

7 Procedure

7.1 Extraction

If there is sufficient test material duplicate determinations shall be carried out.

Approximately 1 g of the specimen is cut into small pieces (about 1 cm^2) and the mass recorded to the nearest 0,1 mg), in order to ensure sufficient wetting, and is transferred into a Erlenmeyer flask or other contamination free vessels.

For any materials except felt and wool use 100 ml vessels. In the case of felt and wool use 250 ml vessels.

Add 50 ml of acid synthetic perspiration solution (5.1) to the test specimen and stopper. The specimen is shaken briefly by hand in order to ensure a complete wetting of the textile sample. Subsequently, the specimen is shaken for 1 h at (37 ± 2) °C. The shaking frequency is adjusted to 60 cycles per minute in case of a horizontal shaker and to 30 cycles per minute using a circular shaker. Alternatively, stirring with a magnetic stirrer is allowed. Subsequently, filter (4.6) the extract in order to remove small textile particles and fluff.

7.2 Determination of metals

Determination of the metal amounts in the digestion solutions is performed complying with EN 1233, EN ISO 11885, EN ISO 12846, EN ISO 15586, EN ISO 17294-2, EN ISO 17852 or ISO 8288.

Calibration is performed either with calibration solutions in the appropriate matrix or via standard addition. In the case of excessive concentrations, the test solutions may be diluted with dilution solution (5.7) depending on the analytical system.

7.3 Reporting of results

The metal contents in the textile have to be specified in milligram per kilogram (mg/kg). The results of 7.2 are calculated according to the following equation:

$$d = \frac{(\beta \cdot V)}{(m \cdot 1000)}$$

where

d metal content as mass portion, in milligrams per kilogram (mg/kg);

β mass concentration of metal in the extraction solution, in micrograms per litre (µg/l);

V volume of solvent, in millilitres (ml);

m weight of specimen, in grams (g).

If a dilution has been applied for testing, the dilution factor has to be taken into account for the determination of the result.

8 Reliability of the method

The results of the round robin test can be found in Annex A.

NOTE "Trueness" and "precision" are terms complying with ISO 5725-1 to describe the accuracy of test methods.

9 Test report

The test report shall state at least the following particulars:

a) name of the laboratory;

b) reference to this European Standard;

c) specification of the applied analytical system;

d) specification of the specimen;

e) amount of each targeted metal in mg/kg;

f) any discrepancy from this European Standard, if relevant.

Annex A
(informative)

Reliability of the method

A.1 Trueness complying with ISO 5725-4

"Trueness" refers to the closeness of agreement between the arithmetic mean of a large number of test results and the true or accepted reference value.

Trueness was calculated as systematic measurement error (bias) of the method and in correlation to the mean value, because no reference value of the investigated material is available.

In a round robin test 2006 of NMP a textile specimen was tested for metal content using different test methods. The measured values of the participating laboratories deviated partly from each other. For result evaluation particular laboratory values ("outliers") were eliminated resulting in good correlation based on the calculation of standard deviation.

Calculation of mean value and standard deviation after elimination of deviating values (marked in grey) for the determination of metals after extraction with acid synthetic perspiration solution.

Table A.1 — Results of statistical evaluation: Determination of contents after extraction with perspiration solution

Metal	**Metal content** mg/kg								**Mean value**	**Standard deviation**	**Variation coefficient**
	Laboratory								*x*	*s*	*v*
	A	**B**	**C**	**D**	**E**	**F**	**G**	**H**	mg/kg	mg/kg	%
Sb	4,59	6,50	6,27	6,77	5,54	7,80	4,30	5,11	5,86	1,189	20
As	0,60	<10	0,46	0,42	0,53	0,45	0,73	0,59	0,54	0,109	20
Pb	0,48	0,30	0,89	0,48	0,44	0,48	0,59	0,96	0,58	0,230	40
Cd	0,07	0,20	0,16	0,20	0,17	0,17	0,17	0,20	0,18	0,018	10
Cr	2,90	2,00	2,94	2,31	2,39	2,91	2,90	3,14	2,69	0,399	15
Co	0,98	1,00	1,14	1,12	1,29	1,29	1,30	1,38	1,19	0,149	13
Cu	6,05	8,10	7,87	9,01	8,72	9,77	10,10	10,00	8,70	1,360	16
Ni	3,56	5,40	6,63	5,41	5,51	6,59	5,90	6,69	6,02	0,602	10
Hg	0,07	0,14	0,06	0,07	0,02	0,03	0,10	0,11	0,09	0,028	31

Table A.2 — Estimation of method trueness: calculation of the systematic deviation (bias) δ of the measurement method complying with ISO 5725-4

Metal	Systematic deviation (bias) δ							
	Laboratory							
	A	B	C	D	E	F	G	H
Sb	–1,27	0,64	0,41	0,91	–0,32	1,94	–1,56	–0,75
As	0,06		–0,08	–0,12	–0,01	–0,09	0,19	0,05
Pb	–0,10	–0,28	0,31	–0,10	–0,14	–0,10	0,01	0,38
Cd	–0,11	0,02	–0,02	0,02	–0,01	–0,01	–0,01	0,02
Cr	0,21	–0,69	0,25	–0,38	–0,30	0,22	0,21	0,45
Co	–0,21	–0,19	–0,05	–0,07	0,10	0,10	0,11	0,19
Cu	–2,65	–0,60	–0,83	0,31	0,02	1,07	1,40	1,30
Ni	–2,46	–0,62	0,61	–0,61	–0,51	0,57	–0,12	0,67
Hg	–0,02	0,05	–0,03	–0,02	–0,07	–0,06	0,01	0,02

Table A.3 — Calculation of the percental recovery rate (extraction yield) for perspiration extraction in reliance to total digestion

Metal	Percental recovery rate (extraction yield) %								Mean value	Standard deviation
	Laboratory								x	s
	A	B	C	D	E	F	G	H	%	%
Sb	40,87	57,88	55,83	60,28	49,33	69,46	38,29	45,50	52,18	9,90
As	96,77		74,19	67,74	85,48	72,58	117,74	95,16	87,10	16,27
Pb	33,33	20,83	61,81	33,33	30,56	33,33	40,97	66,67	41,07	15,70
Cd	36,84	105,26	84,21	105,26	89,47	89,47	89,47	105,26	85,71	21,37
Cr	86,05	59,35	87,24	68,55	70,92	86,35	86,05	93,18	79,71	11,06
Co	69,01	70,42	80,28	78,87	90,85	90,85	91,55	97,18	83,63	9,83
Cu	55,10	73,77	71,68	82,06	79,42	88,98	91,99	91,07	79,26	11,59
Ni	49,72	75,42	92,60	75,56	76,96	92,04	82,40	93,44	79,77	13,49
Hg	50,00	100,00	42,86	50,00	14,29	21,43	71,43	78,57	59,18	24,06

A.2 System precision

System precision correlates with data specified for analytical systems in EN 1233, EN ISO 11885, EN ISO 12846, EN ISO 15586, EN ISO 17294-2, EN ISO 17852 or ISO 8288.

A.3 Limit of detection and limit of quantification

Detection and quantification limits correlate with data specified for the particular measurement systems in EN 1233, EN ISO 11885, EN ISO 12846, EN ISO 15586, EN ISO 17294-2, EN ISO 17852 or ISO 8288.

Annex B
(informative)

Extraction recovery

The absolute contents of the manufactured reference material were determined by total digestion. The extraction recoveries were investigated after the first extraction with acidic artificial perspiration solution (see [1]). The determination of metals was performed by ICP-OES.

Table B.1 — Extraction recovery of reference material

Metal	Extraction recovery %
As[a]	90
Cd	80
Hg[b]	33
Cu	90
Cr	14
Co	80
Ni	81
Pb	11

[a] Measurement with hydride AAS.

[b] Measurement with cold vapor technique.

Bibliography

[1] Thesis, ”Determination of heavy metals in textiles after total digestion in comparison with eluate method. Evaluation of the results with regard to eco specifications“ (A. Hübscher, polytechnic school Hamburg – scientific technology, October 1998

[2] ISO 5725-1, *Accuracy (trueness and precision) of measurement methods and results — Part 1: General principles and definitions*

[3] ISO 5725-4, *Accuracy (trueness and precision) of measurement methods and results — Part 4: Basic methods for the determination of the trueness of a standard measurement method*

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